Improved Cellulose Yield in the Production of Dissolving Pulp from Bamboo Using Acetic Acid in Prehydrolysis

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Despite increasing demand for dissolving pulps from raw material, production costs remain quite high compared to regular paper pulp. Research literature to date has focused on improving performance and quality but has not simultaneously explored how to improve yield, which typically falls below 35%. Dissolving pulp from bamboo, as a widely available, high-quality raw material, was investigated with dilute acetic acid pre-hydrolysis before cooking and cold caustic extraction prior to bleaching. It was found that dilute acetic acid in the pre-hydrolysis stage could speed up the degradation of hemicellulose in bamboo and improve the diffusion of the cooking liquor in bamboo fiber compared with hot-water treatment. The dissolving pulp from bamboo was produced with 93% α-cellulose, 90% ISO brightness, 959 mL/g viscosity, 5.23% xylans, and 0.17% ash. The overall yield on the basis of raw material was 37%, which was higher than other documented processes using other agents in pre-hydrolysis.

Keywords: Bamboo; Dissolving pulp; Acetic acid pretreatment; Yield

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INTRODUCTION

Dissolving pulp is used as a raw material in the manufacture of various cellulose-derived products and textile fibers (Behin and Zeyghami 2009; Patrick 2011). The demand for dissolving pulps in the market during the last 10 years may be attributed to a consistent growth in rayon demand, particularly in China, where 61% of the current global rayon production capacity is located. The annual dissolving pulp production in 2011 was 4.2 million tons, comprised of 2.9 million tons for commodity applications such as rayon and 1.3 million tons converted to specialties such as cellulose acetate (Sixta *et al.* 2013). This trend of increasing demand for dissolving pulps is anticipated to continue over the ensuing decades.

Though the manufacture of dissolving pulp is commercially viable, the overall fiber line yield for dissolving pulp production rarely exceeds 35% and normally retains around 30% (Zhou 2011), which was also in keeping with surveying mills in China before the project. Thus, the production cost of dissolving pulp is quite high compared to regular paper pulp. Unfortunately, to date there has been a scarcity of literature on dissolving pulp yield, despite its relation to the cost of the pulp. Too much emphasis has been placed on the performance of the products, the purity-grade dissolving pulp (Borrega *et al.* 2013; Luo *et al.* 2014), and/or the structures of fiber (Testova *et al.* 2014). Studies are required that simultaneously focus on how to improve the yield of dissolving pulp while retaining cellulose performance.

Bamboo has been demonstrated to be a good source for dissolving pulp (Salmela *et al.* 2008). It can be classified generally as a long-fibered fibrous material, and chemical compositions in many bamboo species are comparable to that of hardwood fibers. Because bamboo is abundant and has good quality of fiber, the technology for converting bamboo chips into dissolving pulp has been attracting increasing attention, particularly given its potential in bamboo textile fiber production.

Pulps used in textile fiber production differ from those used in paper manufacture. Bleached pulp with high purity is free of hemicellulose and lignin, in which the content both of ash and metal ion and degree of polymerization should be strictly controlled (Jahan 2009; Ma 2012). To remove as much hemicellulose, lignin, and other impurities as possible while retaining a high yield of pulp, it is critical to explore a proper process and optimize the technology.

In general, pre-hydrolysis prior to the kraft pulping process is necessary to produce dissolving pulp with a satisfactorily high cellulose and low hemicellulose content. This treatment could also open up the cellulose fibers for further reactions and increase the extractability of lignin during the subsequent pulping process (Lavarack *et al.* 2000; Garrote *et al.* 2003). Procedures currently used in pretreatment include hot-water pre-hydrolysis, wet oxidation (Arvaniti *et al.* 2012), steam pretreatment (Brownell and Saddler 1987), and ammonia fiber explosion (Lee *et al.* 2010). Acetic acid has also been used as a pre-hydrolysis reagent, which is regarded as an environmentally friendly way to obtain cellulose pulps from lignocellulosic materials. Compared with dilute inorganic acid, it has some desirable advantages, including effective hydrolysis, fewer degradation products, and more oligomeric sugars (Kootstra *et al.* 2009).

In the current study, bamboo chips were evaluated as raw material for dissolving pulp production with acetic acid pre-hydrolysis and an alkaline pulping process. The chips were hydrolyzed in dilute acetic acid liquor and subsequently cooked by the kraft pulping process. The pulp was bleached to full brightness with an elemental chlorine-free bleaching sequence, and a cold caustic extraction stage was inserted before the first chlorine dioxide reaction to lower the pulp hemicellulose content. The final yield and compositions of the pulp were evaluated, demonstrating that this is a viable technology for use in the process of dissolving pulp production from bamboo.

EXPERIMENTAL

Materials

Raw materials and pre-hydrolysis

The bamboo chips were generously provided by Nanping Forestry Mill (Zhangzhou City, Fujian, China). The pre-hydrolysis of bamboo was carried out in an M/K digester equipped with a heat exchanger, circulating pump, and computer-controlled time and temperature. The cooking conditions were as follows: 500 g chips, 5 L/1 kg liquid/fiber ratio, maximum temperature 160 °C, 90 min to maximum temperature, 15 min at maximum temperature, and 2.5% acetic acid on a dry weight basis.

Methods

Kraft cooking and bleaching

The kraft cooking of treated chips was performed under the following conditions: 20% effective alkali (EA) and 25% sulfidity on dry chip weight, 5 L/1 kg liquor/bamboo ratio, 60 min to a maximum temperature of 163 °C, and 30 min at top temperature. The pulp was bleached to full brightness with O-CCE-D-(EP)-D sequences, where O refers to the single-stage oxygen delignification, CCE to the cold caustic extraction, D to the chlorine dioxide bleaching, and (EP) to oxidative extraction reinforced with hydrogen peroxide. Table 1 shows the conditions used for each bleaching stage.

Table 1. General Bleaching Conditions

Conditions	O	CCE	D	(EP)	D
Consistency (%)	10	10	10	10	10
Temperature (°C)	105	40	60	80	80
Time (min)	70	60	40	90	90
Pressure (kPa)	600	-	-	-	-
ClO_2 (kg/t)	-	-	10	-	30
NaOH (kg/t)	20	80	-	10	5
$MgSO_4$ (kg/t)	1.5	-	-	-	-
H_2O_2 (kg/t)	-	-	-	3	-

Evaluation of the hydrolyzate and pulps

Pulps were tested according to the Standard Methods of the Technical Association of the Pulp and Paper Industry. The hydrolysis, cooking, and bleaching yields were measured gravimetrically. Solid content of treated liquor was figured out on the basis of weight before and after the evaporating of liquid at 105 °C. Pulp obtained was characterized for their kappa number, viscosity, alpha-cellulose, and brightness, according to TAPPI T236 om-06 (2006), T230 om-08 (2008), T203 cm-99 (1999) and T525 om-06 (2006), respectively. The dissolved lignin in the hydrolyzate was measured based on the UV/Vis spectrometric method at wavelength 205 nm (T 222 om-11 (2011)). Total extractive, ash, acid insoluble lignin, holocellulose and pentosan were measured according to TAPPI T264 cm-07 (2007), T211 om-93 (1993), T222 om-02 (2002), T9wd-75 (1975), and T223cm-10 (2010) respectively.

RESULTS AND DISCUSSION

Characterization of Raw Material and Pre-Hydrolysis

The chemical composition of bamboo is listed in Table 2. In general, non-woods differ somewhat from woods in their chemical composition and properties, which in turn has a direct influence on the pulping and bleaching process. As can be seen from Table 2, the lignin and holocellulose content of bamboo was similar to those in wood, with holocellulose and xylan contents at 72.5% and 23.7%, respectively. This feature renders bamboo interesting in the production of dissolving pulp, given the potential use of extracted xylan and xylan derivatives for biorefinery purposes.

Table 2. Chemical Composition of Bamboo Material

Component	Weight percent on dry basis（%）
Holocellulose	72.5
Lignin	25.2
Pentosan	23.7
Alcohol-benzene Extract	4.23
Ash	1.44

An additional feature of note is the bamboo's content of 4.23% alcohol-benzene extractives and 1.44% ash, which may cause severe difficulties for the utilization of dissolving pulp for production of cellulose derivatives if not properly removed.

The pre-hydrolysis before cooking can degrade some components, such as hemicellulose and extractives from the raw materials and let them dissolve in pretreatment liquor to obtain final pulp with less contaminants. Gray and Casebier (1983) prepared dissolving pulp from southern pine and black Bakelite, and found that the pre-hydrolysis of raw materials was significantly affected by the pre-hydrolysis temperature; that is, under the higher temperature the reaction occurred more quickly and resulted in more dissolved components in the liquor.

When the raw materials are pre-treated with water, the xylan chains crack and the acetyl groups are removed due to the hydrolysis reactions catalyzed by hydronium ions from water auto-ionization. In the later stages of pre-hydrolysis, autoionization of acetic acid produced from acetyl groups provided additional hydronium ions that could improve the pre-hydrolysis kinetics (Heitz *et al.* 1986). Based on the above mechanism, researchers considered how to add additional acetic acid to water during pretreatment to accelerate the hydrolysis. Hemicelluloses tend to be degraded more easily than cellulose by adding acetic acid during pre-hydrolysis (Allen *et al.* 1996). Thus, the cellulose in raw materials was retained and shows improved susceptibility to further reaction in cooking (Garrote and Parajó 2002).

The bamboo chips were treated with acetic acid at different temperatures. The influence of temperature on the removal of both lignin and hemicellulose is shown in Table 3. When pre-hydrolysis was carried out below 160 °C, only a slight loss in raw material mass (less than 10%) took place. Higher temperature led to greater loss in mass. The yields of remaining feedstock after treatment at 170 and 180 °C were 81% and 74%, respectively. To avoid mass loss, Batalha *et al.* (2012) investigated the auto-hydrolysis of bamboo chips at 170 °C, finding that the residual solid yield was 95.1%, which did not sufficiently change plant tissues. The bamboo chips was treated with acetic acid from 0 to 3%, and there was not much change of releasing xylose from 1.5% to 3% so that 2.5% acetic acid was chosen for the prehydrolysis. Acetic acid (2.5% on feedstock) was added to enhance the hydrolysis reaction, and it was possible to achieve the similar yield even at 140 °C as that of autohydrolysis. The result supported the argument that dilute acid condition accelerated the hydrolysis reaction, thus attaining an ideal result at a lower temperature (Rudie *et al.* 2007; Hu *et al.* 2010).

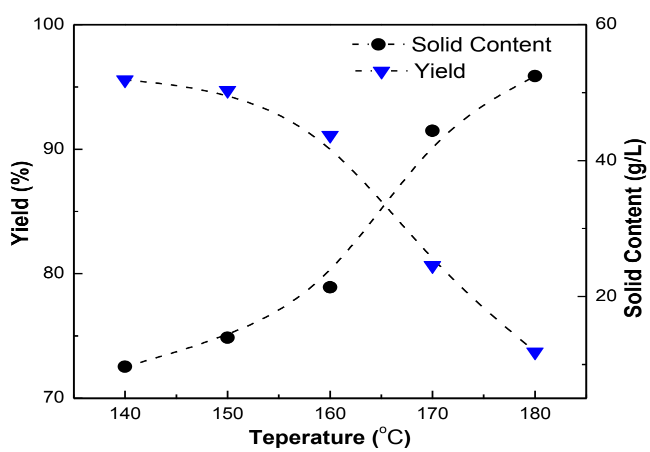

Fig. 1. The hydrolysis yield and solid content in the hydrolyzate at various temperatures

The solid content of treated liquor was increased from 9.68 g/L to 52.44 g/L when the temperature was increased from 140 to 170 °C (Fig. 1). Pre-hydrolysis at 170 °C caused 20% mass loss (Table 2), which may represent a certain degradation of both hemicellulose and cellulose (Li *et al.* 2010). However, with the removal of hemicellulose, increasing amounts of cellulose were exposed to the alkali in the subsequent pulping process, causing cellulose loss and thus lower pulp yield. Thus, to gain a high yield of dissolving pulp, it is necessary to control the hydrolysis reaction and seek a balance during the subsequent process. The temperature, 160 °C was selected to be the reasonable temperature.

Table 3. Influence of Temperature on Pre-hydrolysis* of Bamboo with Acetic Acid

Temperature (°C)	Yield (%)	Xylan removal (%)	Lignin in Hydrolyzate (g/L)	Hydrolyzate pH
140	95.6	4.8	3.68	3.47
150	94.7	5.6	4.10	3.48
160	91.1	10.7	5.65	3.43
170	80.7	27.2	6.81	3.12
180	73.7	61.7	7.89	2.79

*Pre-hydrolysis conditions: ratio of liquor to solid 5:1; concentration of acetic acid 2.5%; time to maximum temperature: 90 min; time at maximum temperature 15 min

Cooking Process

Cooking time directly influenced pulping yield and pulp quality (Table 4). Extending the cooking time may result in the loss of yield, viscosity, and kappa number. It was found that a cooking time of 30 min is reasonable for the pulping process of bamboo. The screened pulp yield determined on the basis of the hydrolyzed chip weight was 45%, which approximated results found by Vu (2004) and Germgård *et al.* (2010). If the cooking time was less than 30 min, the amount rejected was high, which is not beneficial for pulping. Pre-hydrolysis also refers to lignin removal during subsequent kraft pulping, presumably due to better accessibility and the cleavage of LLC bonds (Schild *et al.* 1996; Rauhala *et al.* 2011). Thus, milder cooking conditions, expressed in lower H-factor and chemical charges, were sufficient to achieve the requested degree of delignification and less cellulose loss. The H-factor of the kraft pulping in this study was 325. The overall yield, including acetic acid pre-hydrolysis and kraft pulping, was 41% based on raw materials, which was higher than the yield (<35%) of most work (Lei *et al.* 2008; Ma *et al.* 2012) with bamboo as raw feedstock. The high cooking yield guaranteed a favourable final yield of bleached dissolving pulp. At the same time, the kappa number of 17.4, intrinsic viscosity of 1342 mL/g, and brightness of 27.4% were reasonable enough to produce dissolving pulp with an acceptable level of quality.

Table 4. Kraft Pulping Results for Acetic Acid Hydrolyzate Bamboo Chips*

Cooking Time (min)	H-factor	Yield on Pre-hydrolysis Bamboo (%)	Yield on bamboo (%)	Kappa No.	Intrinsic Viscosity (mL/g)	Brightness (% ISO)
30	325	45	41	17.3	1342	27.3
60	580	44	40	15.1	1329	27.7
90	840	43	39	14.1	1290	24.9
120	1090	43	39	14.4	1232	23.6

*Cooking conditions: Effective alkali 20%; sulfidity 25% (on NaOH); time heating to maximum temperature (from 80 °C to 163 °C) 60 min

Oxygen Delignification and Bleaching

The oxygen delignification efficiency of pulp derived from acetic acid hydrolyzed bamboo chips was 69.78% (Table 5). This was calculated on the basis of the kappa number before and after the O-stage. Viscosity only dropped from 1342 mL/g to 1128 mL/g, which generally matched the results of Mân Vu (2004) and Salmela *et al.* (2008). The pulp yield of the O-stage was 94.55%, indicating that there was little loss of cellulose during this stage. Kraft pulps with a high cooking yield and good viscosity were easily delignified by oxygen delignification to a low kappa number (7 to 9) without any significant loss (110 to 200 mL/g) in viscosity (Mân Vu 2004; Salmela *et al.* 2008).

Table 5. Oxygen Delignification Performance for Pulp Derived from Bamboo

Oxygen Delignification Results	Percentage (%)
Kappa drop	69.8
Viscosity drop	15.9
Brightness out	32.1
Oxygen Delignification Yield	94.6

The oxygen delignification pulps were further bleached to over 90% ISO brightness with the CCE-D-(EP)-D sequences (Table 6). The CCE stage was included in the bleaching process to address the low hemicellulose content, because the efficiency of cellulose conversion into specific derivatives was dependent upon the hemicellulose content of the dissolving pulp (Christov *et al.* 1998). Meanwhile, hemicellulose could also induce numerous of other drawbacks such as poor mechanical properties or yellowing. In the present study, a bamboo bleached pulp only contained 5.23% xylan, which was acceptable for many dissolving pulp applications. The D-(EP)-D sequence was used in the bleaching process, in which the total ClO_2 charge was 4%. The α-cellulose content in the final pulp was 93%, which was suitable for viscose grade pulps (Ibarra *et al.* 2010). At the same time, the viscosity of pulp decreased from 1110 mL/g to 959 mL/g.

The ash content of the bleached bamboo pulp was 0.17%. Recent literature found that ash content for viscose grade bamboo pulp was 0.22% (He *et al.* 2008). This indicated that the proposed acetic acid hydrolysis, cooking, and bleaching method, including a CCE stage, was effective in removing the ash from the bamboo chips.

It is significant that the overall yield of dissolving pulp from bamboo raw material was 37%, which exceeded the general reported level of 30 to 35%. Clearly, this method of producing dissolving pulp from bamboo with acetic acid pre-hydrolysis is of potential significance in commercial production.

Table 6. Properties of Dissolving Pulp after Bleaching

Conditions	O	CCE	D_0-(E_P)-D_1
Kappa Number	5.2	-	-
Brightness (% ISO)	40.3	39.9	90.2
Viscosity, mL/g	1128	1110	959
Alpha-Cellulose (%)	-	-	93.26
Xylans (%)	-	-	5.23
Ash (%)	-	-	0.17
Yield (%)	95	98	98
Overall yield=37%			

Note: - no determination

CONCLUSIONS

1. The substitution of dilute acetic acid for sulfuric acid was effective in pre-hydrolysis of dissolving pulp production from bamboo. The utilization of dilute acetic acid can lower the reaction temperature required to reach a satisfactory hydrolysis of raw material compared to hot water pre-hydrolysis.
2. Due to less cellulose loss during the acetic acid pre-hydrolysis the yield of dissolving pulp from bamboo was as high as 41% after kraft cooking, and the final yield of fully bleached pulp was 37% with good brightness. This exceeds the highest rates previously reported, which is significant for manufacturing mills of dissolving pulp.
3. The conditions of the kraft pulping and O-CCE-D-(EP)-D bleaching sequence proved suitable for the production of dissolving pulp from bamboo. The bleached pulp with 93% α-cellulose, 90% ISO brightness, 959 mL/g viscosity, 5.23% xylans, and 0.17% ash was appropriate for viscose rayon production.

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